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AD-A241 547

DOCUMENTATION PAGE

Form Approved
OMB No 0704-0188

1b RESTRICTIVE MARKINGS	

3 DISTRIBUTION / AVAILABILITY OF REPORT
Approved for public release: distribution unlimited.

2b. DECLASSIFICATION / DOWNGRADING SCHEDULE

4 PERFORMING ORGANIZATION REPORT NUMBER(S)	5. MONITORING ORGANIZATION REPORT NUMBER(S)
G. L. Report No. 4863	

6a. NAME OF PERFORMING ORGANIZATION	6b OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION
Stanford University		Office of Naval Research

6c. ADDRESS (City, State, and ZIP Code)	7b. ADDRESS (City, State, and ZIP Code)
Edward L. Ginzton Laboratory Stanford University Stanford, California 94305-4085	Physics Division, Code 1112 Arlington, VA 22217-5000

8a. NAME OF FUNDING / SPONSORING ORGANIZATION	8b. OFFICE SYMBOL (If applicable)	9 PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
		N00014-90-J-1635

8c. ADDRESS (City, State, and ZIP Code)	10 SOURCE OF FUNDING NUMBERS			
	PROGRAM ELEMENT NO. N63375	PROJECT NO. 4126952	TASK NO	WORK UNIT ACCESSION NO.

11. TITLE (Include Security Classification)
TUNNELING ACOUSTIC MICROSCOPY   UNCLASSIFIED

12. PERSONAL AUTHOR(S)
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13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15 PAGE COUNT
Technical Performance	FROM 04/01/90 TO 03/31/91	1991 September	15

16. SUPPLEMENTARY NOTATION

17	COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB-GROUP	Near-field acoustic microscope micromaching techniques
20	01		Tetrahedral silicon tips gold films
			Piezoelectric transducer tip-sample spacing

19 ABSTRACT (Continue on reverse if necessary and identify by block number)

We have integrated silicon micromachining techniques with piezoelectric thin film deposition to make a near-field acoustic microscope. A piezoelectric zinc oxide (ZnO) transducer is deposited on a substrate of 7740 glass. A sharp tip is formed in a silicon wafer which is anodically bonded to the glass substrate. A sample is attached to substrate of glass with a receiving ZnO transducer. The transducer on the tip excites an ultrasonic beam which passes from the tip to the sample and is detected by the receiving transducer. A feedback signal is generated to keep the transmitted amplitude constant as a sample is raster scanned. The feedback signal is applied to a tube scanner and is also used to modulate the intensity of a display monitor. We find that the instrument has a vertical height sensitivity of about 20 Å, and a lateral resolution of better than 800 Å.

20 DISTRIBUTION / AVAILABILITY OF ABSTRACT	21 ABSTRACT SECURITY CLASSIFICATION
☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT ☐ DTIC USERS	UNCLASSIFIED
22a NAME OF RESPONSIBLE INDIVIDUAL L. E. Hargrove, ONR Physics Division	22b TELEPHONE (Include Area Code) (703) 696-4221	22c OFFICE SYMBOL ONR Code 1112

DD Form 1473, JUN 86   Previous editions are obsolete.   SECURITY CLASSIFICATION OF THIS PAGE

91-11835

Tunneling Acou tic Microscopy

Technical Performance Report

April 1, 1990 - March 31, 1991

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G. L. Report No. 4863

September 1991

Edward L. Ginzton Laboratory
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Tunneling Acoustic Microscopy

Technical Performance Report

April 1, 1990 - March 31, 1991

Our aim for the first year was to build a tunneling acoustic microscope and demonstrate the ability to transmit sound through a tip significantly sharper than the acoustic wavelength and perhaps measuring on atomic dimensions. Our goal is to image mechanical and height variations in a sample. Another aim is to develop an understanding of the mechanisms that allow the transmission of sound through atomic tips. We have demonstrated the ability to transmit through a sharp tip and are in the process of improving the signal-to-noise ratio of the microscope signal in order to carry out two-dimensional scans of samples.

SYSTEM CONFIGURATION

The near-field acoustic microscope consists of an acoustic transducer and tip assembly which together form an acoustic source smaller than the acoustic wavelength (Fig. 1). A sample is imaged by scanning the tip in a raster fashion and measuring the transmitted acoustic signal. A feedback system keeps the transmitted acoustic signal constant by changing the spacing between the tip and the sample. Using this technique we can image both conducting and insulating samples using acoustic impedance and height variations as contrast mechanisms.
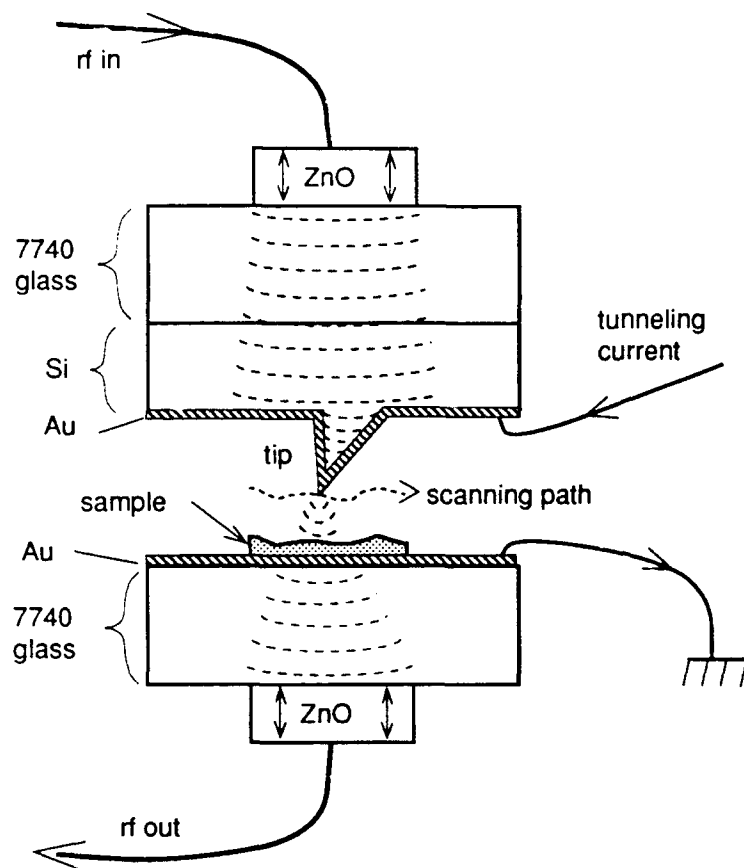
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Fig. 1 Schematic diagram of transducer-tip assembly.

Construction of the near-field acoustic microscope is divided into two distinct categories: the mechanical instrument and the microfabricated tip/transducer assembly. The mechanical instrument and control electronics are identical to those used in STM and described elsewhere.[1,2,3] Our instrument utilizes a tripod design[4] which allows control of separation between the tip and the sample as well as their relative tilt (Fig. 2).
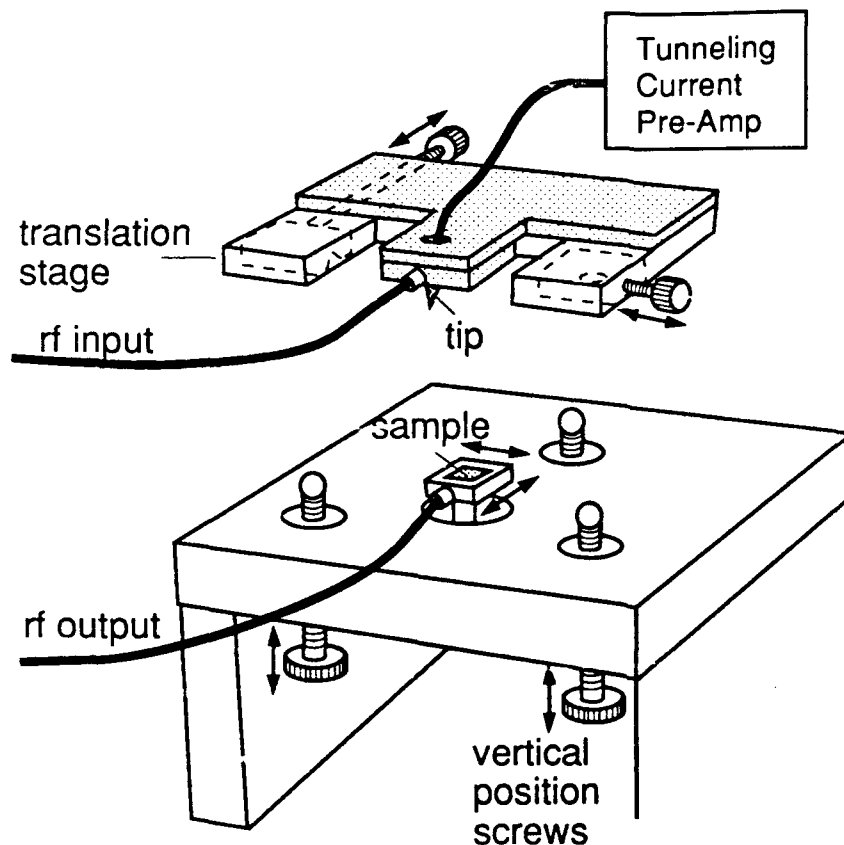
Fig. 2 Schematic diagram of the mechanical scanning system.

The essential feature of the microfabricated tip/transducer assembly is a sharp silicon tip integrated with an ultrasonic transducer. While several methods for fabricating sharp tips are known,[5,6] we use a tetrahedral, single crystal silicon tip. The fabrication process for the tetrahedral tips is outlined in Fig. 3. The process begins with the formation of vertical-walled silicon posts using dry etching. A nitride covered silicon wafer is used so that the resulting post is capped with nitride while its sidewalls are bare silicon. The post is shaped so that at least one sharp corner is pointing in the [110] direction of the wafer. Utilizing a local oxidation of silicon (LOCOS) process, several thousand angstroms of oxide are then grown at low temperatures (950° C) so that the sidewalls of the post are

protected by oxide whereas the top of the post is still capped by nitride. The nitride cap is selectively removed using a reactive ion plasma of SF_6 and CF_3Br. The exposed silicon in the center of the post is then etched in an anisotropic silicon etchant such as EDP or KOH. Most of the post's interior is etched away during this step with the exception of small tetrahedral volumes in the corners of the post. These tetrahedral volumes are not etched since they are bounded on their vertical sides by oxide and on one face by a (111) crystallographic plane. A finished tip is shown in Fig. 4.
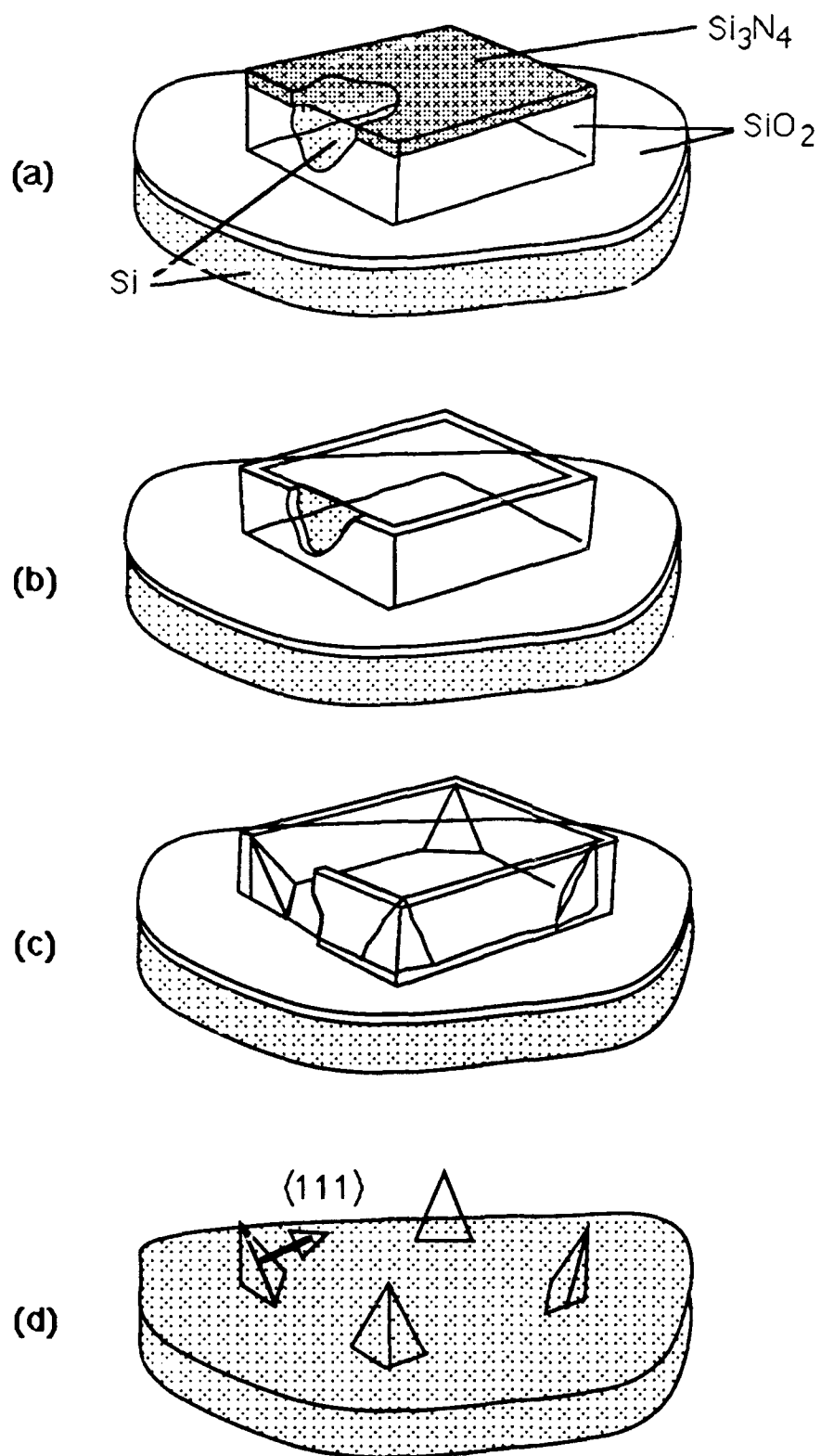
(a)

Si₃N₄

SiO₂

Si

(b)

(c)

(d)

⟨111⟩

Fig. 3   Process outline for making tetrahedral silicon tips.
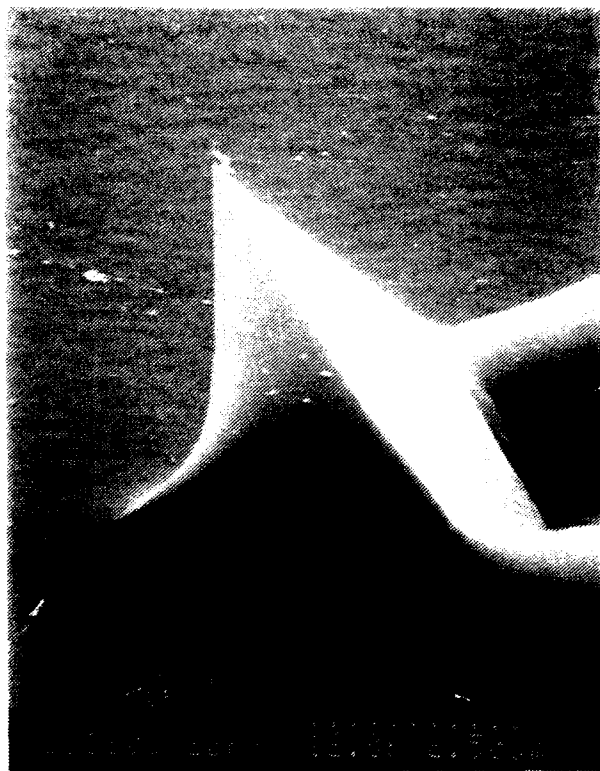
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Fig. 4   A tetrahedral silicon tip.

We favor this tip fabrication technique because it is quite easy to execute. There are no critical, time-dependant etches nor exotic materials to consider. Tips made in this way have radii of curvature typically less than 500 Å and as low as 200 Å. A key factor in the sharpness of these tips is the low temperature oxidation step which is inherent in the process. The relatively high stress in the low temperature oxide causes sharpening of silicon features. Thus, even though the original silicon post has corners whose sharpness

is limited by the resolution of the lithography, the final sharpness is determined by the sharpening effect of the low temperature thermal oxidation step.[6,7]

The ultrasonic transducer uses a sputtered zinc oxide (ZnO) film as the piezoelectric element. The ZnO is sandwiched between two Cr/Au and Ti/Au electrodes forming a 50 x 200 µm transducer with a resonant frequency at 175 MHz. The resonant frequency is determined by the thickness of the ZnO and Au films. The transducers were electrically tuned to operate at a frequency of 135 MHz. The substrate for the transducer is Corning 7740 glass, chosen to be compatible with silicon during a subsequent anodic bonding step.

After fabrication, the tip and the transducer are aligned using a two-sided aligner. A special jig protects the tip at all times. The aligned pieces are then heated to 325° C on a hot plate and anodically bonded for 15 to 30 minutes with 1500 -2500 V applied.

MEASUREMENT SYSTEM

The electronic measurement system combines a superheterodyne detection system with the scanning and height control electronics of an STM. An rf tone burst at the frequency of operation is amplified and applied to the transmitting transducer and tip which is brought into contact with the sample. An identical transducer under the sample detects the tone burst. This transmitted signal is then amplified, gated, mixed with a local oscillator, passed through a diode detector and fed into a signal averager to improve the signal-to-noise ratio. Typically, the transmitting transducer is excited with a 1 watt peak power. The total dynamic range of our system excluding the signal averager is 120 dBm. The one-way insertion loss of a transducer is typically 14 dBs. The efficiency of the transducer can be improved by another 8 dBs by proper electrical tuning, and by paying more attention to the deposition of the ZnO. We are presently working to reduce the insertion loss of the transducer to about 6 dBs.

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The transmitted acoustic signal is used in a feed back loop to control the Z-motion of a cylindrical piezoelectric scanner, and also to modulate the intensity of a display monitor. Thus, images are generated by scanning the tip over a sample and using the feedback signal to modulate the intensity of the display monitor at the location of the tip. The contrast in the images is due to changes in the detected signal caused by both acoustic impedance and height variations.

EXPERIMENTAL RESULTS

The experiments are carried out by placing a sample on a bare ultrasonic receiver which is identical to the tip transducer except for the lack of a tip. We have used 1000 Å evaporated gold films on Corning 7740 glass as a sample as well as an anodically bonded silicon grating. The sample is mounted on a piezoelectric tube scanner commonly used in STMs.[8] The tip transducer is mounted in the microscope head and is held firmly by a kinematic seating arrangement.

In order to align the transmitting tip transducer to the receiving sample transducer, water is first placed in the gap between the tip and sample. A 135 MHz pulsed signal is applied to the tip transducer and the tip is moved in X and Y to maximize the transmitted signal. The amplitude of the transmitted signal is also highly sensitive to tilt so tilt is also optimized by using the tripod screws. When the transmitted signal has been maximized, the water in the gap is removed and the tip is approached to the sample.

Two simple experiments have been conducted. The first was to measure the transmitted signal intensity versus gap spacing and the second was to scan the sample under the tip to try to obtain a topographic image of the sample surface. The first experiment was carried out by putting a modulation of approximately 1000 Å on the sample height. The sample was moved toward and away from the tip at 1 to 10 Hz while the

transmitted signal was monitored. At large gap spacings, no signal was detected. Upon approaching the tip to the sample, the transmitted signal increased in correspondence to the tip position. Figure 5 shows a clear knee in the curve where the transmission begins and increases as the tip is brought closer to the sample At these signal levels, we can detect a 20 Å excursion in the gap spacing at a unity signal-to-noise ratio.
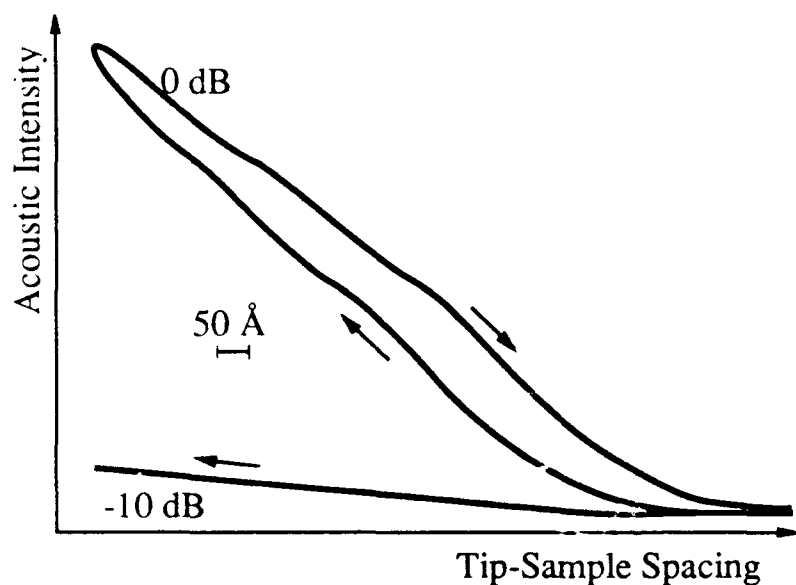

Fig. 5 Transmitted acoustic signal intensity plotted versus tip/sample separation.

A one-dimensional topograph of the glass surface of the sample transducer was obtained by scanning the sample under the tip using height feedback to maintain constant transmitted signal intensity (Fig. 6). The upper curve shows a 4000 Å scan of a region and the lower scan shows the same region scanning a range twice as small For a particular tip these scans are reproducible and repeated scans over the same region show the topography is unchanged. There is significant variation from tip to tip however and degradation in tip performance is observed after hard crashes into the sample.
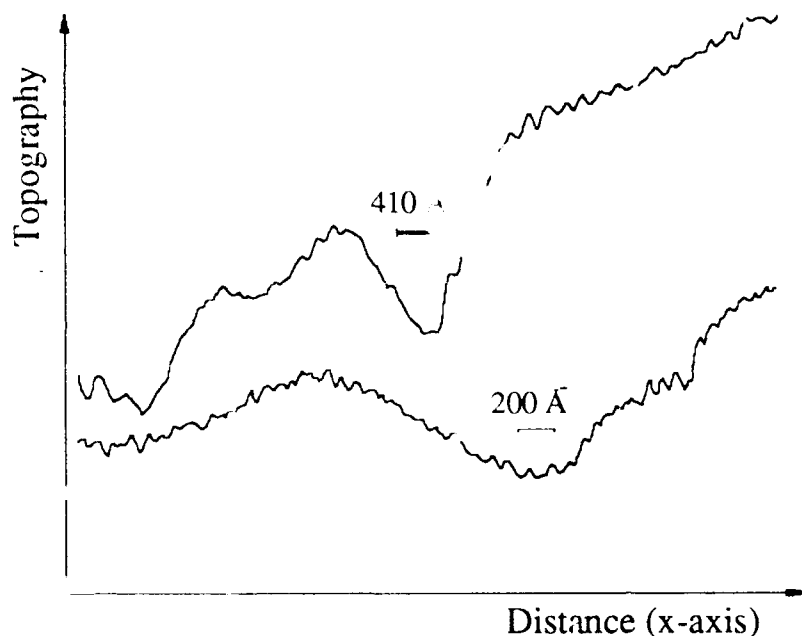
Fig. 6 One-dimensional topography of gold film on glass imaged using feedback
to maintain constant transmitted signal intensity.

Our initial attempts at two-dimensional scans have failed because of the low signal-to-noise ratio in the detected signal. This is a temporary set back as we expect to improve the electrical tuning of the transducer and the mechanical alignment fixture to allow us to succeed in imaging samples. We have already made samples with 6.5 μm gratings in silicon for imaging with the microscope.

DISCUSSION

The results of these experiments indicate that it is possible to transmit acoustic waves from one transducer to another through a point junction. Using an excitation of 135 MHz results in wavelengths on the order of 60 μm in silicon. Nonetheless we are transmitting this signal through a tip with radius of curvature of hundreds or perhaps a few thousand angstroms.

The actual mode of transmission is as yet unclear. Since the experiments were all conducted in air, it is assumed that all surfaces have a contamination layer which may be as thick as a few hundred angstroms. This soft contamination layer may partially explain the seemingly long range interaction of the tip and sample. If the tip were simply crashing into the surface repeatedly, we would expect the location of the knee of the curve as well as the curve's slope to change over time. Instead, we observe that a given tip can be approached over 50 times and the curve does not change significantly. Variations in the curves are seen for different tips.

The one-dimensional scanning data is very interesting since it seems to show a lateral resolution of approximately 1000 Å. While this data was being taken, several parameters such as scan size, rotation, and frequency were varied to eliminate the possibility of scan artifacts.

Our goals for the next year are to demonstrate two-dimensional imaging and to develop a better understanding of the mechanisms that control the performance of the microscope.

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1. "Acoustic Microscopy Beyond the Diffraction Limit: an Application of Microfabrication", S. Akamine, B. Hadimioglu, B.T. Khuri-Yakub, H. Yamada and C. F. Quate - presented at TRANSDUCERS '91, 6th International Conference on Solid-State Sensors and Actuators, San Francisco, CA June 23-27, 1991 - paper given by S. Akamine

2. "Near Field Acoustic Microscopy", B.T. Khuri-Yakub, S. Akamine, B. Hadimioglu, H. Yamada and C. F. Quate - invited paper presented at SPIE Conference on Scanning Microscopy Instrumentation, part of SPIE International Symposium on Optical Applied Science and Engineering - San Diego, CA. July 21-26, 1991 - paper given by B. T. Khuri-Yakub